PATENT ABSTRACTS OF JAPAN

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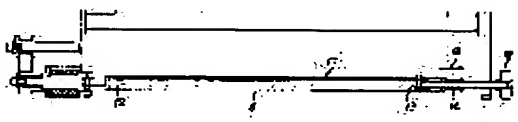
(54) FORMATION OF HIGH-TENSILE FILM ON GRAIN ORIENTED SILICON STEEL SHEET AND FORMING DEVICE

(57)Abstract:
PROBLEM TO BE SOLVED: To efficiently coat a steel sheet with an Si series ceramics film with high reproducibility and to effectively improve its core loss characteristics by executing film formation to the surface and back face of a silicon steel sheet substrate under the same conditions while optimum tensile strength is applied thereto.
SOLUTION: In a vacuum chamber, a DC magnetron sputtering device having a silicon target and a manget is set, and Si ions are vapor-deposited on a silicon steel sheet fixed to the inside of a sample holder via the opening and shuttering of a shutter. The lower end part of the sample holder 6 is provided with a fixing pin 12, and the upper end part is provided with a moving pin 13. A nob 15 provided with a screw groove 14 is rotated to move the moving pin 13 in the direction of the arrow 16, the interval between the fixing pin 12 and the moving pin 13 is expanded, and tensile strength is applied to the silicon steel sheet substrate 17. The sample holder 6 is rotated by 180° by the operation of a handle provided at the outside of a tank, and film formation is executed to the surface and rear face of the substrate 17 under the same conditions. The additional tensile strength for the substrate 17 is preferably controlled to about 0.1 to 5.0 kg/mm2.



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[Claim(s)]

[Claim 1] A tension coat formation method of a grain-oriented magnetic steel sheet characterized by moreover forming the surface and a rear face on the same conditions, adding tension to a silicon steel substrate in on the other hand forming Si system ceramic film on the surface of tropism silicon steel by DC magnetron spatter in a vacuum tub which introduced reactant gas.

[Claim 2] In claim 1, addition tension to a silicon steel substrate A tension coat formation method of a grain-oriented magnetic steel sheet characterized by being 0.1 - 5.0 kg/mm2.

[Claim 3] While forming a magnetron sputtering system which offers a silicon target and a magnet in a vacuum tub which introduced reactant gas A sample electrode holder which counters this silicon target and holds a silicon steel substrate is prepared. This sample electrode holder A silicon target is received by actuation from the outside of a tub. While having a rolling mechanism in which 180-degree rotation is possible Tension coat formation equipment of a grain-oriented magnetic steel sheet characterized by having a tension addition device which adds tension to this substrate by pulling a held silicon steel substrate at the both ends.

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention uses much more improvement in an iron loss property as a drawing wax by moreover forming the surface and a rear face under the same conditions, adding proper tension to a silicon steel substrate forming the ceramic film of Si system especially on the occasion of the surface of a grain-oriented magnetic steel sheet about the tension coat formation method and formation equipment of a grain-oriented magnetic steel sheet by the so-called magnetron spatter.

[0002]

[Description of the Prior Art] The ion plating method using the plasma is applied to ceramic coating, such as TiN, TiC, and Ti (CN). as the ion plating method -- HCD -- law

and EB (Electron Beam)+RF (Radio Frequency) Technique, such as law, the Marti arc process, and an arc discharge method, is enforced. the inside of such technique -- especially -- HCD -- law -- an ionization rate -- 20 - 40% -- high -- moreover, membrane formation speed -- 0.05-0.5 mum/min Since it is comparatively quick, it is widely used for ceramic coating, such as TiN, TiC, Ti (CN), or CrN. Moreover, even if requirements, such as pretreatment of N2 quantity of gas flow, a degree of vacuum, bias voltage, substrate temperature, and a substrate, change a little, there is also an advantage that ceramic coating can be performed easily and smoothly in this HCD method.

[0003] In addition, recently, the method of forming a reactant ceramic film using a magnetron spatter has also come to be performed briskly. this method -- ** -- a film uniform to ** cross direction in which high-speed membrane formation is comparatively possible is obtained -- ** When **DD (direct current) magnetron spatter is used, there is not only an advantage -- the coating film which vacuum evaporationo effectiveness is good and carried out ** long duration stability is obtained -- but The membrane formation to the substrate which has large surface area is also comparatively easy, and high-speed membrane formation is possible, and it is pointed out that the outstanding reactant coating films (for example, Si system ceramic film etc.) are obtained.

[0004] Although use of the HCD method described above about the steel plate which has the large surface area used for construction material etc. since it was an improvement of corrosion resistance, fanciness, or abrasion resistance is tried, it has not resulted by utilization the place which it is till the present. because, it mentioned above -- as -- HCD -- although the membrane formation speed of law is large -- 0.05-0.5 mum/min a degree -- batch type coating -- enough -- coming out -- it is because it is inadequate for applying to coating of large surface area of a certain thing, and it is necessary to raise the ionization rate of the quality of an emission more, and to perform high-speed membrane formation

[0005] advanced features of the surface controlling method by the ceramic coating on such the surface of a steel plate -- in addition, if a TiN ceramic film is covered on the surface of tropism silicon steel on the other hand, powerful **** tension will be given near the silicon steel surface recently -- a 180" magnetic domain subdivides extremely -- **** -- it was found out as a result that silicon steel shows super-low iron loss. Thus, coating of a TiN ceramic film attracts attention as what shows a possibility of manufacture of a product of having a new highly efficient material.

[0006] However, also in manufacture of such a highly efficient material, re-evaluation of coating material and a coating method is obliged from a viewpoint of product quality or a manufacturing cost. That is, although super-low iron loss was obtained when a TiN ceramic film was covered on the surface of tropism silicon steel on the other hand, it had

left the problem at the place which says that a manufacturing cost is expensive. For example, when TiN was covered to silicon steel using the HCD method, it had the big defect that the coating cost of TiN became expensive, for that about [that Ti material is expensive] and vacuum evaporationo effectiveness is as low as about 20%, the price of Ta cathode being high, etc.

[0007] In order that this TiN covering might conquer the defect of the another very recently above, it was found out also by using DC (direct current) magnetron spatter and on the other hand covering Si system ceramic (Si-N-O-C) on the surface of tropism silicon steel that silicon steel shows super-low iron loss. Remarkable high-speed membrane formation is not only possible for covering of Si system ceramic (Si-N-O-C) film on the surface of silicon steel which used this DC magnetron spatter, but it has become clear that vacuum evaporationo effectiveness is good, that a film uniform crosswise is obtained, for coating which carried out long duration stability to be possible, etc.

[0008]

[Problem(s) to be Solved by the Invention] Processing area aims [this invention] at what the large tension coat formation method which can coat with Si system ceramic film often [repeatability] and efficiently to tropism silicon steel on the other hand, and can raise effectively the magnetic properties, especially the iron loss property of silicon steel is used for operation of the method, and is proposed with suitable tension coat formation equipment using the above-mentioned DC magnetron spatter.

[0009]

[Means for Solving the Problem] That is, this invention is the tension coat formation method for a grain-oriented magnetic steel sheet characterized by forming the surface and a rear face on the same conditions, adding tension to a substrate on the surface of tropism silicon steel on the other hand by DC magnetron spatter in a vacuum tub which introduced reactant gas in carrying out membrane formation processing of the Si system ceramic film.

[0010] Moreover, while this invention forms a magnetron sputtering system which offers a silicon target and a magnet in a vacuum tub which introduced reactant gas A sample electrode holder which counters this silicon target and holds a silicon steel substrate is prepared. This sample electrode holder A silicon target is received by actuation from the outside of a tub. While having a rolling mechanism in which 180-degree rotation is possible It is tension coat formation equipment of a grain-oriented magnetic steel sheet characterized by having a tension addition device which adds tension to this substrate by pulling a held silicon steel substrate at the both ends.

[0011] Addition tension [on this invention and as opposed to a silicon steel substrate] It is desirable to consider as about two 0.1 - 5.0 kg/mm.

[0012]

[Embodiment of the Invention] First, the tension coat formation equipment of the grain-oriented magnetic steel sheet according to this invention is explained. It uses for operation of this invention and a ** type shows suitable tension coat formation equipment to drawing 1 . The number 1 in drawing is a vacuum tub, and can hold the interior now to a high vacuum by suction from the suction opening 2. In this vacuum tub 1, DC magnetron sputtering system which has a silicon target 3 and a magnet 4 (they are three pieces at this example) is installed, and it has become the structure which makes Si ion vapor-deposit through closing motion of a shutter 5 to the silicon steel fixed in the sample electrode holder 6.

[0013] By equipping the structure of this sample electrode holder 6 with the feature, and operating the handle 7 prepared out of the tub by this invention, a pinion 8 is minded and it is about the sample electrode holder 6. It is the structure which can be rotated 180 degrees. In addition, as for a rack and 10, 9 is [a sample electrode-holder case and 11] heaters.

[0014] Although drawing 2 shows the longitudinal section of this sample electrode holder 6 By rotating the knob 15 which a lock-pin 12 is formed in the lower limit section of the sample electrode holder 6, and the migration pin 13 is formed in the upper limit section on the other hand, and formed the screw slot 14 That is [it moves the migration pin 13 in the direction of an arrow head 16], by extending the gap of a lock-pin 12 and the migration pin 13, it is the structure which can add tension to these pins 12 and the silicon steel substrate 17 fixed by 13. On the other hand, it is actuation of a handle 7 in which the sample electrode holder 6 was formed out of the tub as mentioned above. If this silicon steel substrate 17 is reversed and Si system ceramic film is similarly -ed **(ed) after -ed **(ing) Si system ceramic film on the surface of the silicon steel substrate 14, since it is the structure which can be rotated 180 degrees, it will be the reason which can form the surface and a rear face on the same conditions.

[0015] In addition, addition tension to a silicon steel substrate when -ed **(ing) Si system ceramic film on the surface of silicon steel using above equipment It is desirable to consider as about two 0.1 - 5.0 kg/mm. Because, addition tension 0.1 kg/mm2 It is because it will come to exceed the elasticity limit of silicon steel and the fall of flux density will be caused, if it is deficient in the effect if it does not fill, and 5.0 kg/mm2 is exceeded on the other hand. Moreover, among drawing 1 , although Mark L is the distance between the silicon steel substrates 17 held at the silicon target 3 and the sample electrode holder 6, it is designed with this invention equipment, so that this

distance L can be displaced freely.

[0016] Next, Si system ceramic film is formed on the surface of silicon steel using the above-mentioned tension coat formation equipment, and the result investigated about the magnetic properties acquired in that case is explained.

C: 0.072 wt% and Si:3.39wt% and Mn:0.076 wt%, Mo: 0.013 wt% and Se:0.021 wt% and aluminum:0.020 wt%, Sb: 0.026 wt% and N:0.0071wt% was contained and the remainder used as the last cold-rolled board of 0.23mm thickness the silicon steel hot-rolling board (thickness: 2.0 mm, width-of-face:1200mm) which becomes the presentation of Fe substantially with two cold rolling which sandwiches 1050-degree C intermediate annealing. To the surface of silicon steel, it is width-of-face:200 after that. mum, the depth: 20 micrometers The slot was introduced in the direction of a right angle at intervals of 4mm to the rolling direction. Then, after giving decarbonization and primary recrystallization annealing in ****** of 840 degree C, Since MgO (25%), aluminum 2O3 (50%), ZnO (3%), and the annealing separating medium that uses SiO2 (20%) and TiO2 (2%) as a principal component are applied 15h secondary recrystallization annealing is performed at 850 degrees C. Subsequently, after developing secondary recrystallization grains which carried out the temperature up to 1080 degrees C by h in 12 degrees C /and which were accumulated on Goss bearing, purification annealing was given in 1220-degree C dry hydrogen.

[0017] Subsequently, the oxide on the steel plate surface is removed and it is 0.05 micrometers at the center line average of roughness height by electrolytic polishing. It finished. Then, the tension coat formation equipment shown by the ** type is used for drawing 1 , and it is Si system ceramic (Si-N-O-C system) film. 0.5 micrometers It thickness (per one side) -ed **(ed). In addition, grant tension [as opposed to a silicon steel substrate at this time] 2.0 kg/mm2 It carried out.

[0018] When investigated about the magnetic properties of the product obtained in this way, the result shown below was obtained.

flux density B8 = 1.92 T iron loss W17/50 = 0.48 W/kg -- the case where Si system ceramic (Si-N-O-C system) film is formed at the table rear face of silicon steel in this way using the tension coat formation equipment according to this invention -- the magnetic properties of silicon steel, especially iron loss property W17/50 Having decreased to 0.48 W/kg attracts attention.

[0019] Thus, the extremely excellent iron loss property is acquired by moreover carrying out ceramic coating of the surface and the rear face of silicon steel on the same conditions, adding the suitable tension for silicon steel using the tension coat formation equipment of this invention.

[0020]

[Example] Example 1C: 0.074 wt% and Si:3.43wt% and Mn:0.078 wt%, Mo: 0.012 wt% and Se:0.020 wt% and aluminum:0.020 wt%, Sb: 0.025 wt% and N:0.0077wt% was contained and the remainder used as the last cold-rolled board of 0.23mm thickness the silicon steel hot-rolling board (thickness: 2.2 mm, width-of-face:1200mm) which becomes the presentation of Fe substantially with two cold rolling which sandwiches 1050-degree C intermediate annealing. Subsequently, it is width-of-face:200 to the surface of a cold-rolled board. mum, the depth: 20 micrometers The slot was introduced in the rolling direction and the direction of a right angle at intervals of 4mm. Then, after giving decarbonization and primary recrystallization annealing in ****** of 840 degree C MgO (25%), aluminum 2O3 (50%), ZnO (3%), and the annealing separating medium that uses SiO2 (20%) and TiO2 (2%) as a principal component are applied. Occasion After performing 15h secondary recrystallization annealing at 850 degrees C and developing secondary recrystallization grains which carried out the temperature up to 1080 degrees C by h in 12 more degrees C./and which were accumulated on Goss bearing, purification annealing was given in 1220-degree C dry hydrogen.

[0021] Subsequently, the oxide on the steel plate surface is removed and it is 0.05 micrometers at the center line average of roughness height by electrolytic polishing. It finished. Then, DC magnetron sputtering system shown in the mimetic diagram of drawing 1 is used, and it is Si system ceramic (Si-N-O-C system) film. 0.6 micrometers It thickness (per one side) -ed **(ed). In addition, grant tension [as opposed to a silicon steel substrate at this time] 1.5 kg/mm2 It carried out. The result investigated about the magnetic properties of the obtained product is shown below.

Flux density B8 = 1.92 T iron loss W17 / 50 = 0.49 W/kg [0022]

[Effect of the Invention] In this way, the extremely excellent iron loss property can be acquired by moreover carrying out ceramic coating of the surface and the rear face on the same conditions, adding the suitable tension for a silicon steel substrate according to this invention.

[Brief Description of the Drawings]

[Drawing 1] It is the mimetic diagram of the tension coat formation equipment of a grain-oriented magnetic steel sheet according to this invention.

[Drawing 2] It is the detail drawing of a sample electrode holder.

[Description of Notations]

1 Vacuum Tub

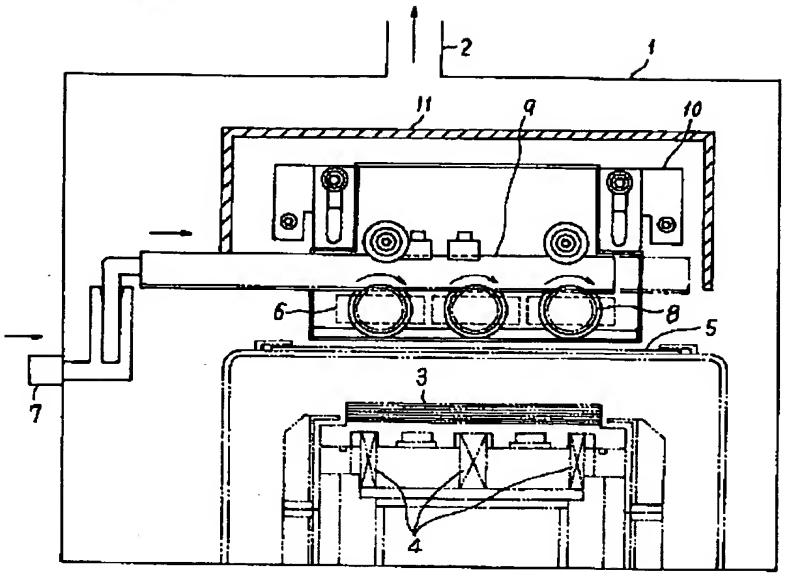
2 Suction Opening

3 Silicon Target

4 Magnet

5 Shutter
6 Sample Electrode Holder
7 Handle
8 Pinion
9 Rack
10 Sample Electrode-Holder Case
11 Heater
12 Lock-pin
13 Migration Pin
14 Screw Slot
15 Knob
16 The Direction of Hauling
17 Silicon Steel Substrate

[Drawing 1]



[Drawing 2]